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Determination of $CaCO_3$ Content in Egg Shell

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Abstract
The complexometry titration investigation has been conducting to ascertain the CaCO3 concentration of Egg. The complex substance EDTA will hold the calcium, and the point will be signaled by a change in the metallochromic indicator's color. Standardization EDTA : Pipette 25 mL of MgSO4.7H2O solution, 10 mL of buffer solution pH 10, a little amount of EBT indicator, and aqua DM into the titration flask. When you titrate with EDTA solution, the solution turns blue. Data processing has revealed that eggshell contains 0,025 grams of calcium and that the calcium content is 14,1%. Theoretically, eggshell is made up of crystals of CaCO3 (98,43%) based on the current mineral composition. Ca3(PO4)2 (0.75%) and MgCO3 (0.84%). Calcium carbonate is the main component of eggshell. When the titrant and titrate interact to create a complex molecule, the titration is known as a complexometric titration. A solution containing Ca2+ will form a complex with EDTA, according to the fundamental tenet of complexometry. CaCO3 was present in the sample (duck eggshell) in an amount of 0.025 grams, and a CaCO3 value of 15.7% was determined.

INTRODUCTION

Egg shells are the most abused household waste. Currently, egg shells are only used as a raw material for craftsmanship. Each egg has 10,000 to 20,000 pores which are thought to absorb solutes and be used as adsorbents.

The formation of the chicken egg part is influenced by the food eaten by the mother. The first is egg wrapping known as eggshell. This egg shell requires calcium and phosphorus as well as vitamin D in its formation. Lack of minerals and vitamins will cause abnormalities in the mother, child and eggs. The average weight of an egg shell is about 5 grams and 40 percent is calcium. Most of the calcium in the egg shell precipitates within 16 hours. Calcium is supplied by special bone masses found in chicken bones that accumulate large reserves of calcium for shell formation. If a hen is fed a low-calcium diet, the eggshell becomes thinner, and the hen can use 10% of the total calcium in her bones just to form an egg. If the feed is persistently low in calcium, egg production will eventually stop. Usually, the raw materials are Ca2+ ions and CO3 2- ions, supplied by the blood to the shell glands. (Syam, 2016)

Chemists have studied the nutritional content of eggshells. The eggshell consists of 95.1% inorganic matter and 3.3% protein 1.6% water. The chemical composition of the egg shell is 1.71% protein, 0.36% fat and water. 0.93%, crude fiber 16.21%, ash 71.34%. Based on the results of previous studies, according to Miles, eggshell powder contains as much as 401 ± 7.2 grams of calcium or about 39% of calcium in the form of calcium carbonate. There is also strontium 372 ± 161 µg, toxic substances such as Pb, Al, Cd and Hg are present in small quantities, as well as V, B, Fe, Zn, P, Mg, N, F, Se, Cu and Cr. Dry eggshell contains about 95% calcium carbonate and weighs 5.5 grams. (Zulfitri,2014)

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The most common method for analyzing calcium levels is using the titirimetry method. Another method used for calcium analysis is the atomic absorption spectroscopy method using ICP-AES (Inductively Coupled Plasma Atomic Emission Spectroscopy). In fact, atomic absorption spectroscopy and titrimetry are widely used because they are simpler, more accurate and have higher precision. However, these equipments are special and few are owned by testing laboratories in Indonesia and are relatively expensive, making it difficult to apply when routine analysis is required. so as a cheaper alternative, the titrimetric method can be used. The titrimetric method used for calcium analysis is complexometric. (Rollando, 2019)

Complexometric titration is a method of titration based on the formation of a complex between a multivalent metal ion and a water-soluble ligand. Compounds that have two lone pairs of electrons are called ligands. The metal ions that react are metal ions where the d subscale is not completely filled. The complex formation reaction is a Lewis acid-base reaction. The commonly used complex forming agent is ethylene diamine tetraacetic acid (EDTA) which will form a strong complex with a ratio of 1:1 with the metal.

Complexometric titrations using edetate disodium require an alkaline pH and a bufferto ensure that the freed protons do not decrease the pH. Support that can be used is an ammonia solution that buffers up to a pH of about 10. Disodium edetate is a stable compound and water-dissolved, which gives the sharp end point and best of all, reacts with Most metal ions are in a 1:1 ratio regardless of the valency of the ions. In this way, metal ions such as Zn2+, Ca2+, and Al3+ can be determined in pharmaceutical samples. (Lubis, 2018)

The method also described as permits analytical determination of measurements for a number of multivalent cations in aqueous solution. This method is based on the formation of complexes between multivalent metals and organic chelates which are water soluble and practically do not dissociate. The formation of a chelate is an organic anion which at a certain distance has several groups with the basic function of electrons or organic compounds with two or more electron donating groups at a certain distance. Each molecule will form one or more rings with two or more divalent metal ions. The complex that occurs in this way is called a chelate because of its scissor-like shape. (Cairns, 2008)

MATERIALS AND METHODS

Materials

The tools used in this experiment were 250 ml measuring flask, 25 ml volumetric pipette, 5 ml volumetric pipette, 500 ml beaker, 250 ml beaker, 50 ml burette, 250 ml titration flask or Erlemeyer, 10 ml measuring cup, and also mortals. The materials used in this experiment are 0.05M EDTA solution, MgSO4.7H2O solid, Buffer pH 10, EBT/NaCl indicator, Murexid/NaCl indicator, egg shells from chicken, duck, goose and quail eggs, 6 M HCl solution, and 4 M NaOH solution.

Methods:

For standardization of EDTA solutions we doing: Place 40 mL of 0.05 M EDTA solution in a 500 mL beaker and dilute up to 200 mL. Place this solution into the burette. Accurately weigh the solid $MgSO_4.7H_2O$ (±0.6 gram), then dissolve it in a 250 mL measuring flask. Pipette 25 mL of this solution into a titration flask, add 10 mL of buffer solution pH 10, a small spoonful of EBT/NaCl indicator and aqua DM. Titrate this solution with EDTA solution so that the color of the solution changes to blue. Do a duplo. Then determine the concentration of the EDTA solution correctly.

For determination of CaCO3 content in eggshells we doing: Clean the egg shell from the remaining membrane, if necessary rinse with water. Place the cleaned eggshells in an evaporating dish or watch glass, then dry in the oven at 105°C for 30 minutes. Cool the egg

shells then grind until smooth with a mortar. Weigh carefully ± 3 grams of egg shells that have been mashed and then place into a 250 mL beaker. Add aqua DM and 50 mL of 6 M HCI while stirring gently. Do it in a fume cupboard. Slowly heat the egg shell solution obtained while stirring until it boils most of the solids dissolve, then cool. Filter the solution obtained and then dilute it in a 250 volumetric flask mL. Pipette 25 mL of this sample solution into a 100 mL measuring flask and dilute to boundary sign. Pipette 5 mL of this last solution into an Erlemeyer flask, add 50 mL of aqua DM, 2 mL of 4 M NaOH solution and murexid/NaCl indicator. Titrate with standard EDTA solution until the solution turns purple blue. Do a duplo. Then determine % CaCO3 in eggshells.

RESULTS AND DISCUSSION

Observation Result

Table 1. Observation Result

No	Observation	Reaction
1.	**Standardization of EDTA**	
	EDTA solution Appearance : Liquid Color : Colorless Indicator EBT/NaCl Appearance : Solid Color : Dark / Black MgSO4 + Ind. EBT → Dark purple color of solution Titration with EDTA → The color of solution is change in to blue (TAT)	$MgSO_4^{2+}{}_{(aq)} + H_2Y^{2-}{}_{(aq)} \rightarrow MgY^{2-}{}_{(aq)} + 2H^{+}{}_{(aq)}$ $Mg\ Ind^{-}{}_{(aq)}$ (red) + $H_2Y^{2-}{}_{(aq)} \rightarrow MgY^{2-}{}_{(aq)}$ (Colorless) + $H\ Ind^{-}{}_{(aq)}$ (blue) + $H^{+}{}_{(aq)}$
2.	**Determination of CaCO3 levels in Eggshell**	
	Sample (Eggshell duck) Appearance : Oval Solid (Before grind) Color : White After Grind Appearance : Powder Color : White Sample powder + Aquadest → sample dissolve in aquadest Appearance : Liquid Color : Colorless NaOH solution Appearance : Liquid Color : Colorless Murexid Appearance : Solid Color : Dark Red Sample + Water + NaOH Appearance : Liquid Color : Colorless + Ind. Murexid → the color of solution is red Titration with EDTA Appearance : Liquid Color : Purple-Blue (TAT)	$Ca^{2+}(aq) + H_2Y^{2-}{}_{(aq)} \rightarrow CaY2^{-}{}_{(aq)} + 2H^{+}{}_{(aq)}$

The volume of titration
Volume 1 : 2,6 ml
Volume 2 : 2,4 ml

Calculation

1. **Standarization Of EDTA**

Volume of MgSO4 : 25 ml
Volume of EDTA : 20 ml
Concentration of MgSO4 : 0,0090 M

Concentration Of MgSO4

Concentration MgSO4 $= \frac{mass}{molecular\ mass}\ x\ \frac{1000}{volume}$

Concentration MgSO4 $= \frac{0{,}6\ grams}{264\ g/mole}\ x\ \frac{1000}{250}$

Concentration MgSO4 = 0,0090 M

Concentration Of EDTA

Concentration EDTA $= \frac{Volume\ x\ Concentration\ (MgSO4)}{Volume\ EDTA}$

Concentration EDTA $= \frac{25\ ml\ x\ 0{,}0090\ M}{20\ ml}$

Concentration EDTA = 0,0025 M

FP EDTA $= \frac{V2}{V1} = \frac{200\ ml}{40\ ml} = 5$

2. **Determination CaCO3 In Sample**

Volume sample : 5 ml
Volume titration : 2,3 ml

[CaCO3] $= \frac{V2\ x\ M2}{V1} = \frac{2{,}5\ ml\ x\ 0{,}01125\ M}{5\ ml} = 0{,}005625$ M

Mass of CaCO3 $= \frac{[CaCO3]X\ V\ CaCO3\ x\ Molecular\ mass}{1000} = \frac{0{,}00562\ M\ X\ 5\ ml\ x\ 100\ g/mole}{1000} = 0{,}0028\ grams$

Mass Ca in CaCO3 $= \frac{Ar\ Ca}{Molecular\ mass\ CaCO3}\ x\ weight\ of\ sample$

Mass Ca in CaCO3 $= \frac{40}{100}\ x\ 0{,}0028\ grams = 0{,}001125\ grams$

Mass of Ca in Sample = mass of Ca x $\frac{V\ volumetric}{V\ of\ pipette}$

Mass of Ca in Sample = 0,00125 grams x $\frac{100\ ml}{5\ ml}$

Mass of Ca in Sample = 0,025 grams

$$\% \text{ Ca} = \frac{mass\ of\ Ca\ in\ Egg\ Shell}{Mass\ of\ Sample}\ x\ 100\%$$

$$\% \text{ Ca} = \frac{0{,}025\ grams}{0{,}159\ grams}\ x\ 100\%$$

$$\% \text{ Ca} = 15{,}7\ \%$$

Discussion

In this experiment we do the complexometric titration to determine the CaCO3 that contain in eggshell. We do many treats to eggshell before titrating with EDTA (Ethylen Diamin Tetra Acetic), the egg that we used is duck egg.

Complexometric titration are known as reaction involving the formation of complex ions or the formation of dissociated neutral molecules in solution. The basic requirement for the formation such as complex is a high degree of solubility. The selectivity of the complex can be adjusted by controlling the pH eg Mg, Ca and Cr can be titrated at pH 11. Most complexometric titration use indicator that also acts as a complexes and of course the metal complex has a different color from the complexing itself. The stability of the complex formed depends on the nature of the cation and the pH of solution, therefore titration us carried out at a certain pH in solution that are too alkaline it is necessary to pay attention to the possibility of precipitation of metal hydroxides. Compexometry is also a method of determining the concentration of a metal ion with complex forming. (Tristiunto. 2017)

Before doing titration, we do standardization of EDTA. EDTA is a weak acid with four protons. Titrate with primer solution is MgSO4 and Buffer solution pH 10 .the atmosphere of the solution must be adjusted to pH 10 for this reaction to take place completely in addition, the complexing reaction with EDTA always produces H ions so that the pH 10 to maintain an atsmophere of pH 10, because the atmosphere of the solution is set to pH 10 then it takes a metal indicator that works at pH 10 as well. (Ningsih.2020). EDTA standardization titration process with the reactions :

$$MgSO_4^{2+}{}_{(aq)} + H_2Y^{2-}{}_{(aq)} \rightarrow MgY^{2-}{}_{(aq)} + 2H^{+}{}_{(aq)}$$

The indicators that we used are EBT and murexid. EBT is sensitive to changes in solution pH. And metal content, murexid is metallochromatic indicators that can form complexes with Ca^{2+}. That's why we used these indicators for complexometric titration. (rosvita.2019). If an indicator is added before the titration, the indicators will form a complex with Mg2+ (red color) then the Mg2+ in the complex will react with EDTA then the red color will disappear the a slight excess of EDTA causes the end point to occur, namely the occurrence of a blue color. Reaction :

$$Mg\ Ind^{-}{}_{(aq)}\ (red) + H_2Y^{2-}{}_{(aq)} \rightarrow MgY^{2-}{}_{(aq)\ (Colorless)} + H\ Ind^{-}{}_{(aq)}\ (blue) + H^{+}{}_{(aq)}$$

After doing standardization, we found the concentration actual of EDTA. Where we do duplo for standardization, so we get the 20 ml the volume of EDTA. After doing calculation we get 0,01125 M for the actual concentration of EDTA. After this we do titration to Eggshell. For eggshell, we dry it in oven in 105^{o}C for 2 hours to evaporate the impurity that contain in eggshell. After oven it, we grind the eggshell until being powder. Then, we weigh the sample about 0,0016 grams. The weigh that weighted of our sample is 0,00159 grams. The sample that grind then put in 250 ml beaker glass and adding 10 ml aqua DM and HCl 6 M, stirring slowly. The purpose of solute with HCl is to hydrolysis process and will form the

bubble gas at solution. We do heating process to speed up the reaction. Then we cool the solution where the solution is homogenous.

For determining the CaCO3 used the murexid indicator with titration where EDTA is secunder solution because murexid is metallochromatic indicator that canform complexes with $Ca^{2+.}$ Murexid indicator has a pH 11-13 and the color changes shown is purplish red to purple blue. Before doing titration we add NaOH which purpose is to neutralize and made base atmosphere in solution. The color of purple after titration sample because murexid has a pH 11-13 for EBT at standardization the Mg^{2+} ion will react with EBT metal indicator to form Mg-EBT that color is purple. During titration with EDTA, Mg^{2+} ion will pull up to EDTA than EBT because Mg-EDTA is stronger that Mg-EBT so, EBT back to free condition that's why wu get blue at standardization.

From the result of dara processing, the weigh of Ca in Eggshell is 0,025 grams and the rate of Ca is 14,1 %. Theoritically based on the existing mineral composition, eggshell are composed of crystals of $CaCO_3$ (98,43%). $MgCO_3$ (0,84%) and $Ca_3(PO_4)_2$ (0,75%). (Bata.2018). The largest content eggshell is calcium carbonate, from the data above, the $CaCO_3$ content in duck eggshell sample is low The reaction:

$$Ca^{2+}(aq) + H_2Y^{2-}{}_{(aq)} \rightarrow CaY2^{-}{}_{(aq)} + 2H^{+}{}_{(aq)}$$

we maybe doing a mistake during experiment. We must be detailed when observed the point titration on burette, we must carefully ehen weigh sample and pipette sample, because the small mistake can effect the all result in aur experiment.

CONCLUSIONS

From the experiment for determining the level of $CaCO_3$ in eggshell, which has been carried out, it can be concluded that :

1. Complexometric titration is a type of titration in which the titrant and the titrate react with each other to form a complex compound. The basic principle of this complexometric titration is that a solution containing Ca^{2+} will form a complex with EDTA so that the concentration can be know, with a blue-purple end point titration (TAT).
2. From the result of dara processing, the weigh of $CaCO_3$ in the sample (eggshell duck) was 0,025 grams and it wa obtained that the $CaCO_3$ content in duck eggshell was 15,7%.

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